

CLAIMS

1. A continuous production method of a polyamide, comprising continuously producing a polyamide by melt polymerization using a multistage polymerization reaction apparatus,

wherein a self-cleaning horizontal twin-screw reaction apparatus is used as a final polymerization reaction apparatus constituting the multistage polymerization reaction apparatus,

wherein the final polymerization is effected while performing an operation of purging inert gas inside the final polymerization reaction apparatus or while performing two or three operations selected from the group consisting of an operation of purging inert gas inside the final polymerization reaction apparatus, an operation of vacuating the final polymerization reaction apparatus, and an operation of adding an end group adjusting agent into the final polymerization reaction apparatus, and

wherein the melt viscosity of the polymer is controlled by continuously measuring the melt viscosity of a polymer at an outlet of the final polymerization reaction apparatus by a viscometer and automatically controlling at least one operation amount out of the inert gas purged amount, the vacuum degree and the amount added of the end

group adjusting agent corresponding to said operations so that the measured viscosity value becomes a value within a previously set definite range.

2. The continuous production method of a polyamide as claimed in claim 1, wherein in performing two operations selected from the group consisting of the inert gas purging operation, the vacuum operation and the addition operation of an end group adjusting agent, one operation amount out of two operation amounts is set as a fixed value and the other operation amount is automatically controlled.

3. The continuous production method of a polyamide as claimed in claim 1, wherein in performing all the three operations selected from the group consisting of the inert gas purging operation, the vacuum operation and the addition operation of an end group adjusting agent, two operation amounts out of three operation amounts are each set as a fixed value and only the remaining one operation amount is automatically controlled, or only one operation amount out of three operation amounts is set as a fixed value and the other two operation amounts are automatically controlled.

4. The continuous production method of a polyamide as claimed in claim 1, wherein the inert gas has a moisture percentage of 0.05 wt% or less.

5. The continuous production method of a polyamide as claimed in claim 1, wherein the polyamide comprises an m-xylylenediamine (MXD) as a diamine component, and the m-xylylenediamine (MXD) content is at least 70 mol% based on the diamine component.

6. The continuous production method of a polyamide as claimed in claim 1, wherein a polyamide having a relative viscosity [RV] of 1.6 to 4.0 is obtained.

7. A continuous production method of a polyamide mainly comprising a diamine component unit and a dicarboxylic acid component unit, said method comprising:

(a) a raw material preparation step of individually melting a diamine and a dicarboxylic acid or forming a salt of amine and carboxylic acid in water,

(b) a raw material introduction step of continuously introducing the prepared raw material into a tubular reaction apparatus,

(c) an amidation step of passing the introduced raw material through the tubular reaction apparatus, thereby

effecting amidation to obtain a reaction mixture containing an amidated product and a condensed water,

(d) an initial polymerization step of introducing said reaction mixture into a continuous reaction apparatus capable of separation and removal of water, and elevating the polymerization degree while separating and removing water at a temperature higher than the melting point of the finally obtained polyamide to obtain a polyamide prepolymer, and

(e) a final polymerization step of introducing the polyamide prepolymer into a continuous reaction apparatus capable of separation and removal of water, and further elevating the polymerization degree at a temperature higher than the melting point of the finally obtained polyamide to obtain a polyamide adjusted to a desired relative viscosity [RV].

8. The continuous production method of a polyamide as claimed in claim 7, wherein the tubular reaction apparatus used for the amidation step (c) has L/D of 50 or more, wherein the inner diameter of the tube is D (mm) and the length of the tube is L (mm).

9. The continuous production method of a polyamide as claimed in claim 7, wherein the average residence time in the amidation step (c) is from 10 to 120 minutes.

10. The continuous production method of a polyamide as claimed in claim 7, wherein the shear rate (γ) in the amidation step (c) is 0.1 (1/sec) or more and the shear stress (τ) is 1.5×10^{-5} Pa or more.

11. The continuous production method of a polyamide as claimed in claim 7, wherein in the amidation step (c), the relative viscosity [RV] of the reaction mixture is elevated by 0.05 to 0.6.

12. The continuous production method of a polyamide as claimed in claim 7, wherein the average residence time in the initial polymerization step (d) is from 10 to 150 minutes.

13. The continuous production method of a polyamide as claimed in claim 7, wherein the continuous reaction apparatus in the final polymerization step (e) is a horizontal reaction apparatus.

14. The continuous production method of a polyamide as claimed in claim 7, wherein the continuous reaction apparatus in the final polymerization step (e) is a self-cleaning horizontal twin-screw reaction apparatus.

15. The continuous production method of a polyamide as claimed in claim 7, wherein the average residence time in the final polymerization step (e) is from 1 to 30 minutes.

16. The continuous production method of a polyamide as claimed in claim 7, wherein the relative viscosity [RV] of the polyamide obtained in the final polymerization step (e) is from 1.6 to 4.0.

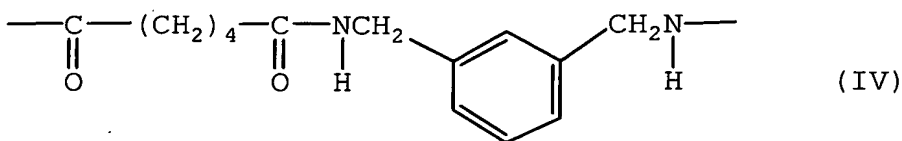
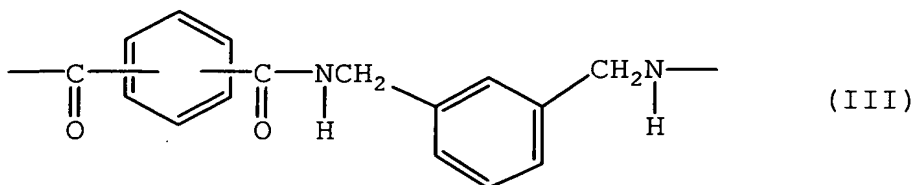
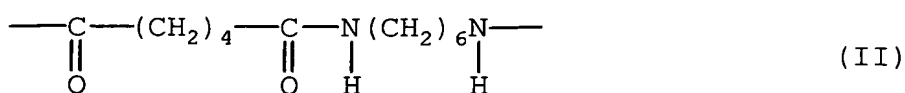
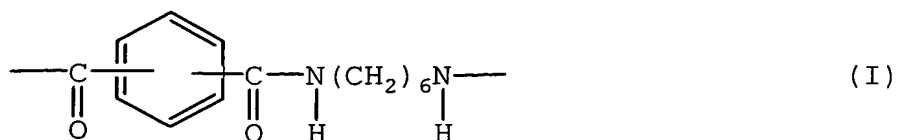
17. The continuous production method of a polyamide as claimed in claim 7, wherein in the final polymerization step (e), the relative viscosity [RV] of the polyamide is controlled by an operation of purging inert gas inside the reaction apparatus, an operation of adjusting vacuum degree in the reaction apparatus, an operation of adding an end group adjusting agent into the reaction apparatus, or a combination thereof.

18. The continuous production method of a polyamide as claimed in claim 7, wherein in the final polymerization step (e), the final polymerization is effected while performing an operation of purging inert gas inside the reaction apparatus or while performing two or three operations selected from the group consisting of an operation of purging inert gas inside the reaction apparatus, an operation of vacuating the reaction apparatus, and an operation of adding an end group adjusting agent into the reaction apparatus, and

wherein the melt viscosity of the polymer is controlled by continuously measuring the melt viscosity of a polymer at an outlet of the final polymerization reaction apparatus by a viscometer and automatically controlling at least one operation amount out of the inert gas purged amount, the vacuum degree and the amount added of the end group adjusting agent corresponding to said operations so that the measured viscosity value becomes a value within a previously set definite range.

19. The continuous production method of a polyamide as claimed in claim 7, wherein in the raw material preparation step (a), the atmospheric oxygen concentration at the preparation of raw material is 10 ppm or less.

20. The continuous production method of a polyamide as claimed in claim 7, wherein the polyamide comprises at least one member selected from the group consisting of the following repeating units (I) to (V):



21. The continuous production method of a polyamide as claimed in claim 19, wherein the polyamide comprises at least one member selected from the group consisting of the repeating units (I), (III) and (IV).

22. The continuous production method of a polyamide as claimed in claim 7, wherein the polyamide comprises an

m-xylylenediamine (MXD) as a diamine component, and the m-xylylenediamine (MXD) content is at least 70 mol% based on the diamine component.

23. A continuous production method of a polyamide mainly comprising a diamine component unit and a dicarboxylic acid component unit, said method comprising:

(a) a raw material preparation step of individually preparing a melted diamine and a melted dicarboxylic acid,

(b) a raw material introduction step of continuously introducing the melted diamine and the melted dicarboxylic acid into a polymerization reaction apparatus to get a diamine and a carboxylic acid together by using raw material supply means comprising a raw material supply device, a mass flow rate measuring device provided on the downstream side of said raw material supply device and a control system of automatically controlling the output of said supply device such that the mass flow rate measured by said mass flow rate measuring device becomes a previously set value, and

a polymerization step of polycondensing the diamine and the dicarboxylic acid introduced into the polymerization reaction apparatus.

24. A continuous production method of a polyamide mainly comprising a diamine component unit and a dicarboxylic acid component unit, said method comprising:

(a) a raw material preparation step of individually preparing a melted diamine and a melted dicarboxylic acid,

(b) a raw material introduction step of continuously introducing the melted diamine and the melted dicarboxylic acid into a tubular reaction apparatus to get a diamine and a carboxylic acid together by using raw material supply means comprising a raw material supply device, a mass flow rate measuring device provided on the downstream side of said raw material supply device and a control system of automatically controlling the output of said supply device such that the mass flow rate measured by said mass flow rate measuring device becomes a previously set value,

(c) an amidation step of passing the diamine and dicarboxylic acid gotten together through the tubular reaction apparatus, thereby effecting amidation to obtain a reaction mixture containing an amidated product and a condensed water,

(d) an initial polymerization step of introducing said reaction mixture into a continuous reaction apparatus capable of separation and removal of water, and elevating the polymerization degree while separating and removing

water at a temperature higher than the melting point of the finally obtained polyamide to obtain a polyamide prepolymer, and

(e) a final polymerization step of introducing the polyamide prepolymer into a self-cleaning horizontal twin-screw reaction apparatus capable of separation and removal of water, and further elevating the polymerization degree at a temperature higher than the melting point of the finally obtained polyamide to obtain a polyamide adjusted to a desired relative viscosity [RV].

25. The continuous production method of a polyamide as claimed in claim 24, wherein the tubular reaction apparatus used for the amidation step (c) has L/D of 50 or more, wherein the inner diameter of the tube is D (mm) and the length of the tube is L (mm).

26. The continuous production method of a polyamide as claimed in claim 24, wherein the average residence time in the final polymerization step (e) is from 1 to 30 minutes.

27. The continuous production method of a polyamide as claimed in claim 24, wherein the relative viscosity [RV]

of the polyamide obtained in the final polymerization step (e) is from 1.6 to 4.0.

28. The continuous production method of a polyamide as claimed in claim 24, wherein in the final polymerization step (e), the relative viscosity [RV] of the polyamide is controlled by an operation of purging inert gas inside the reaction apparatus, an operation of adjusting vacuum degree in the reaction apparatus, an operation of adding an end group adjusting agent into the reaction apparatus, or a combination thereof.

29. The continuous production method of a polyamide as claimed in claim 24, wherein in the final polymerization step (e), the final polymerization is effected while performing an operation of purging inert gas inside the reaction apparatus or while performing two or three operations selected from the group consisting of an operation of purging inert gas inside the reaction apparatus, an operation of vacuating the reaction apparatus, and an operation of adding an end group adjusting agent into the reaction apparatus, and

wherein the melt viscosity of the polymer is controlled by continuously measuring the melt viscosity of a polymer at an outlet of the final polymerization reaction

apparatus by a viscometer and automatically controlling at least one operation amount out of the inert gas purged amount, the vacuum degree and the amount added of the end group adjusting agent corresponding to said operations so that the measured viscosity value becomes a value within a previously set definite range.

30. The continuous production method of a polyamide as claimed in claim 24, wherein in the raw material preparation step (a), the atmospheric oxygen concentration at the preparation of raw material is 10 ppm or less.

31. The continuous production method of a polyamide as claimed in claim 24, wherein the polyamide comprises an m-xylylenediamine (MXD) as a diamine component, and the m-xylylenediamine (MXD) content is at least 70 mol% based on the diamine component.

32. The continuous production method of a polyamide as claimed in claim 24, wherein the relative viscosity [RV] of the polyamide obtained in the final polymerization step (e) is from 1.6 to 4.0.